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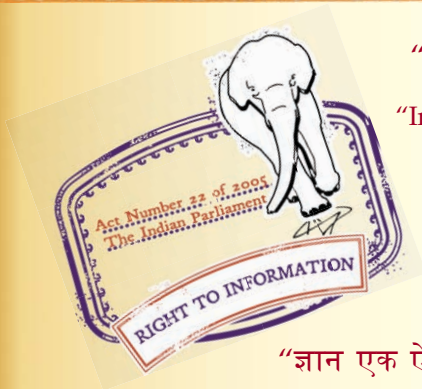
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IS 7696 (1991): Para-cresyl Methyl Ether [PCD 18: Natural and Synthetic Fragrance Materials]



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पैरा-क्रिसाइल मिथाईल ईथर — विशिष्ट  
( पहला पुनरीक्षण )  
*Indian Standard*  
**PARA-CRESYL METHYL ETHER**  
**— SPECIFICATION**  
( *First Revision* )

UDC 665.53 : 547.563.13' 023' 261

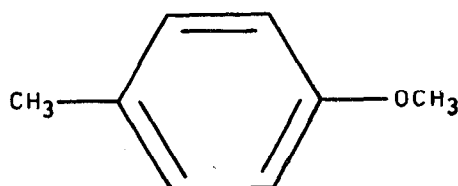
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## FOREWORD

This Indian Standard ( First Revision ) was adopted by the Bureau of Indian Standards, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Para-cresyl methyl ether (  $C_8H_{10}O$  ), also known as methyl para-cresol is usually prepared by the methylation of para-cresol. It is also present in oil of ylang ylang, cananga and others. Its structural formula is as follows.



PARA-CRESYL METHYL ETHER  
( Molecular Mass 122.17 )

It is of considerable importance in perfumery and is used in many floral compositions. It is also used to a limited extent in flavours.

This standard was first published in 1975. At that time due to non-availability of a standardized test procedure for chromatographic analysis of this material, it was decided that GLC method would be incorporated at a later date.

First revision of this standard includes gas chromatographic method of analysis as the main method for determination of purity of para-cresyl methyl ether.

Besides, two new requirements namely minimum purity and peroxide value have been included in this revision.

In the preparation of this standard, considerable assistance has been derived from the following publications :

EOA No. 224-1963 Standard for methyl para cresol. Essential Oil Association of USA, New York.

The Givaudan Index 1961, Ed 2. Givaudan — Delawanna Inc. New York.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in the standard.

# Indian Standard

## PARA-CRESYL METHYL ETHER — SPECIFICATION

( First Revision )

### 1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for para-cresyl methyl ether.

### 2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard :

IS No.	Title
326	Methods of sampling and test for natural and synthetic perfumery materials ( Part 1 ) : 1984 Sampling ( <i>second revision</i> ) ( Part 2 ) : 1980 Preliminary examination of perfumery materials and samples ( <i>second revision</i> ) ( Part 3 ) : 1980 Relative density ( <i>second revision</i> ) ( Part 5 ) : 1986 Determination of refractive index ( <i>second revision</i> ) ( Part 6 ) : 1986 Determination of solubility in ethanol ( <i>second revision</i> ) ( Part 9 ) : 1980 Determination of ester value after acetylation and free alcohols ( <i>second revision</i> )
1070 : 1977	Water for general laboratory use ( <i>second revision</i> )
2284 : 1988	Method for olfactory assessment of natural and synthetic perfumery materials ( <i>first revision</i> )
6597 : 1988	Glossary of terms relating to natural and synthetic perfumery materials ( <i>first revision</i> )

### 3 TERMINOLOGY

For the purpose of this standard, the definitions of terms given in IS 6597 : 1988 shall apply.

### 4 REQUIREMENTS

#### 4.1 Description

Para-cresyl methyl ether shall be a clear colourless liquid, free from sediment, suspended matter, separated water and adulterants.

#### 4.2 Solubility

When tested as prescribed in IS 326 ( Part 6 ) : 1986, the material shall be clearly soluble in 9

volumes of 70 percent ethyl alcohol or 3 volumes of 80 percent ethyl alcohol.

4.3 The material shall also conform to the requirements given in Table 1.

**Table 1 Requirements for Para-Cresyl Methyl Ether**

SI No.	Characteristic	Requirement	Method of Test, Ref to
( 1 )	( 2 )	( 3 )	( 4 )
i)	Colour and appearance	Colourless liquid	IS 326 ( Part 2 ) : 1980
ii)	Odour	Characteristic pungent odour suggestive of ylang ylang, free from objectionable by-odours such as cresylic, etc	IS 2284 : 1980
iii)	Relative density at 27°/27° C	0.964 7 to 0.968 7	IS 326 ( Part 3 ) : 1980
iv)	Refractive index at 27°C	1.5090 to 1.5120	IS 326 ( Part 5 ) : 1986
v)	Free pcresol, percent by mass, <i>Max</i>	2.0	Annex A
vi)	Peroxide value	Nil	Annex B
vii)	Boiling point, °C	175 to 176	Annex C
viii)	Purity, percent by mass, <i>Min</i>	98	Annex D

### 5 PACKING AND MARKING

#### 5.1 Packing

The material shall be supplied in well closed containers, preferably glass, tin-lined, stainless steel or aluminium, as agreed to between the purchaser and the supplier. The material shall be protected from light and stored in a cool and dry place.

#### 5.2 Marking

Each container so filled shall bear legibly and indelibly the following information:

- Name of the material;
- Indication of the source of manufacture
- Batch number and date of manufacture; and
- Net and gross mass.

## 6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 326 ( Part 1 ) : 1984.

## 7 TESTS

7.1 Tests shall be conducted as prescribed in IS 326, IS 2284 : 1988 and Annex A to Annex C. Reference to relevant standards is given in col 4

of Table 1.

## 7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water ( see IS 1070 : 1977 ) shall be employed in tests.

NOTE—'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

## ANNEX A

[ Table 1, Sl No. ( v ) ]

### DETERMINATION OF FREE para-CRESOL

#### A-1 PROCEDURE

A-1.1 Weight accurately about 5 g of sample into a 150-ml glass-stoppered flask. Pipette 10 ml of 10 percent acetic anhydride solution in anhydrous pyridine into the flask. Using the same pipette, transfer 10 ml of acetic anhydride solution into a second flask containing no sample and treat it as a blank. Connect each flask to a water-cooled condenser and reflux for one hour. A guard tube filled with fused calcium chloride should be fixed to the open end of condenser. Discontinue heating the flasks for a few minutes until the temperature of the mixture is below 100°C and then add 25 ml of 50 percent ethyl alcohol-water mixture down the condenser into each flask. Reflux again for 10 minutes to hydrolyse the acetic anhydride. Allow to cool slightly and disconnect the flasks. When the blank and samples are cool, titrate

with 0.5 N potassium hydroxide solution to the end point of phenolphthalein indicator.

#### A-2 CALCULATION

A-2.1 Free para-cresol, percent by mass

$$= \frac{5.407 \times N (V_2 - V_1)}{M}$$

where

$N$  = normality of the potassium hydroxide solution used;

$V_2$  = volume in ml of potassium hydroxide solution used in the blank determination;

$V_1$  = volume in ml of potassium hydroxide solution used in the test sample solution; and

$M$  = mass in g of the sample taken for test.

## ANNEX B

[ Table 1, Sl No. ( vi ) ]

### DETERMINATION OF PEROXIDE VALUE

#### B-1 REAGENTS

B-1.1 Acetic Acid — Analytical reagent grade.

B-1.2 Chloroform

B-1.3 Sodium Thiosulphate Solution — 0.01 N approximately.

B-1.4 Potassium Iodide — saturated solution.

B-1.5 Starch — freshly prepared saturated solution.

#### B-2 PROCEDURE

B-2.1 Weigh accurately about 2 g of the material into a conical flask ( 250 ml capacity ) with provision for stoppering. Add 25 ml of acetic acid chloroform mixture solution ( 3:2 v/v ). Swirl to dissolve the material completely. Add 2 ml of freshly prepared saturated aqueous solution of potassium iodide. Allow the solution to stand with occasional shaking for 1

minute and then add 35 ml of distilled water. Add 2 ml of freshly prepared starch solution. Titrate against sodium thiosulphate ( 0.01 N ). End point being the disappearance of the blue colour. A blank titration using all the reagents but without sample should be carried out.

#### B-4 CALCULATION

B-4.1 Calculate the peroxide value in the material as follows:

Peroxide value, milliequivalents of peroxy oxygen/kg =  $\frac{(V_1 - V_2) \times N \times 1000}{M}$

where

$V_1$  = volume in ml of sodium thiosulphate solution used for the test sample;

$V_2$  = volume in ml of sodium thiosulphate solution used for blank titration;

$N$  = normality of sodium thiosulphate; and

$M$  = mass of the sample in g.

## ANNEX C

### [ Table 1, Sl No. ( vii ) ]

#### DETERMINATION OF BOILING ( DISTILLATION ) POINT/RANGE

##### C-0 OUTLINE OF THE METHOD

**C-0.1** The range of temperature between which a liquid boils or the percentage of the material that distills between two specified temperatures is determined. The lower of the two temperatures is the corrected thermometer reading when the first five drops of distillate have been collected, and the upper temperature is the corrected reading when the percentage specified has been collected.

##### C-1 APPARATUS

###### C-1.1 Distillation Flask

Having a bulb of 50 to 60 ml capacity and a neck 10 to 12 mm long and 14 to 16 mm in internal diameter.

###### C-1.2 Straight Glass Condenser

With a water jacket 40 to 60 cm long, the distance from the upper end of the jacket to the neck of the flask being 18 to 25 cm.

###### C-1.3 Asbestos Board

12 to 15 cm<sup>2</sup> and 3 to 5 mm thick and having a circular perforation, located centrally, to hold the flask. The edge of the perforation shall fit the flask closely when the latter is set into it. The size of the perforation shall be such that when the flask is set into it, the portion of the flask below the upper surface of the asbestos has a capacity of 3 to 4 ml.

###### C-1.4 \*Thermometer

Thermometer conforming to the following requirements is recommended. When placed in position, the top of the bulb of the thermometer

is levelled with the centre of the opening of the outlet tube :

Range	-2°C to +300°C
Graduation	1°C
Immersion	Total
Overall length	380 ± 10 mm
Stem diameter	5.5 to 8.0 mm
Bulb shape	Cylindrical
Bulb length	8 to 16 mm
Bulb diameter	Not less than 5.5 and not greater than stem
Length of graduated portion	223 to 254 mm
Distance from bottom of bulb to 0°C	100 to 110 mm
Longer lines at each	5°C
Figured at each	10°C
Expansion chamber	Required
Top finish	Ring
Scale error not to exceed	±0.5°C up to 150°C ± 1°C above 150°C

Any other thermometer of similar range and accuracy may be used.

##### C-2 PROCEDURE

**C-2.1** Place the asbestos board on a tripod or other suitable support. Transfer to the distillation flask 25 ml of the liquid to be tested, insert the thermometer and place the flask in an up-right position in the perforation of the asbestos board. Connect the distillation flask with the source of heat and distill at the rate of 4 to 5 ml per minute, noting the temperature as soon as 5 drops of the liquid have distilled into the receiver, and when the specified percentage has distilled over.

\*Thermometers with the Institute of Petroleum Designation IP 5C conform to these requirements.

## ANNEX D

### [ Table 1, Sl No. ( viii ) ]

##### D-1 GAS CHROMATOGRAPHIC ANALYSIS OF PARA-CRESYL METHYL ETHER

**D-1.1** The chromatographic conditions given here are for guidance only.

###### D-1.2 Outline of the Method

A sample of the material is dissolved in a suitable solvent namely ethyl acetate, hexane, cyclohexane and chlorocarbon and is injected

into the gas chromatograph, from where it is carried by the carrier gas from one end of the column to the other. During its movement, the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column.



**D-2 APPARATUS**

**D-2.1** Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The typical chromatogram for para-cresyl methyl ether using a chromatograph with the following chromatographic conditions is shown in Fig. 1.

Sample Column	Para-Cresyl Methyl Ether
Material	Stainless Steel
Length	2.43m
OD	0.32 cm
ID	0.20 cm
Stationary phase	FFAP <sup>1</sup> , 10 percent by mass
Solid support	Chromosorb WAP
Carrier Gas	Nitrogen
Conditions	
Column temperature	150°C
Injection port temperature	200°C
Detector	
Type	F.I.D.
Temperature	285

<sup>1</sup> Free fatty acid phase ( FFAP ) in carbowax 20 M treated with nitrophthalic acid

**D-3 CALCULATION****D-3.1 Area Measurement ( see Note 1 )**

Since normal peaks approximate a triangle, the area is measured by multiplying the peak height with the width of half-height. The normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.

**D-3.2 Area Normalization ( see Note 2 )**

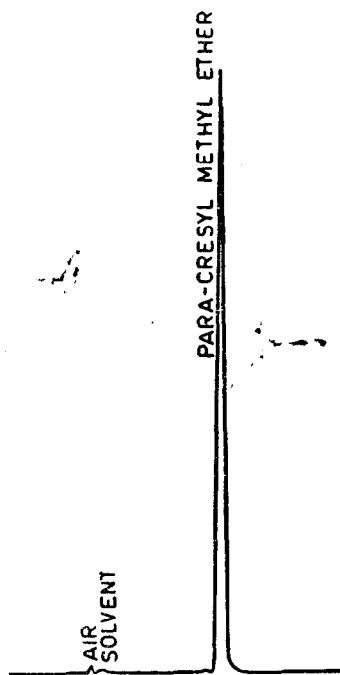
By normalizing, it is meant, calculating the percentage composition by measuring the area of each and dividing the individual areas by total area, for example,

$$\text{Percentage of } A = \frac{\text{Area of } A}{\text{Total area}} \times 100$$

**NOTES**

1 Other methods of area measurements, namely, triangulation, disc integrator and electronic digital integrator, if fixed with GLC machine, would be of great advantage.

2 Internal standardization method may be used if pure appropriate internal standard is available. This method is known as relative or indirect calibration.



**FIG. 1 A TYPICAL CHROMATOGRAM OF  
PARA-CRESYL METHYL ETHER**

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